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(PCT Rule 61.2)

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International filing date (day/month/year) 07 June 1999 (07.06.99)	**Priority date** (day/month/year) 05 September 1998 (05.09.98)
Applicant COLE, Michael	

1. The designated Office is hereby notified of its election made:

   [X] in the demand filed with the International Preliminary Examining Authority on:

   25 February 2000 (25.02.00)

   [ ] in a notice effecting later election filed with the International Bureau on:

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NOTIFICATION OF THE RECORDING OF A CHANGE

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IMPORTANT NOTIFICATION

Date of mailing (day/month/year) 10 October 2000 (10.10.00)	
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1. The following indications appeared on record concerning:

[X] the applicant [ ] the inventor [ ] the agent [ ] the common representative

Name and Address	State of Nationality	State of Residence
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2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:

[X] the person [ ] the name [ ] the address [ ] the nationality [ ] the residence

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3. Further observations, if necessary:
Applicant for all designated States except the United States of America. Mr. Michael Cole is applicant/inventor for the United States of America only.

4. A copy of this notification has been sent to:

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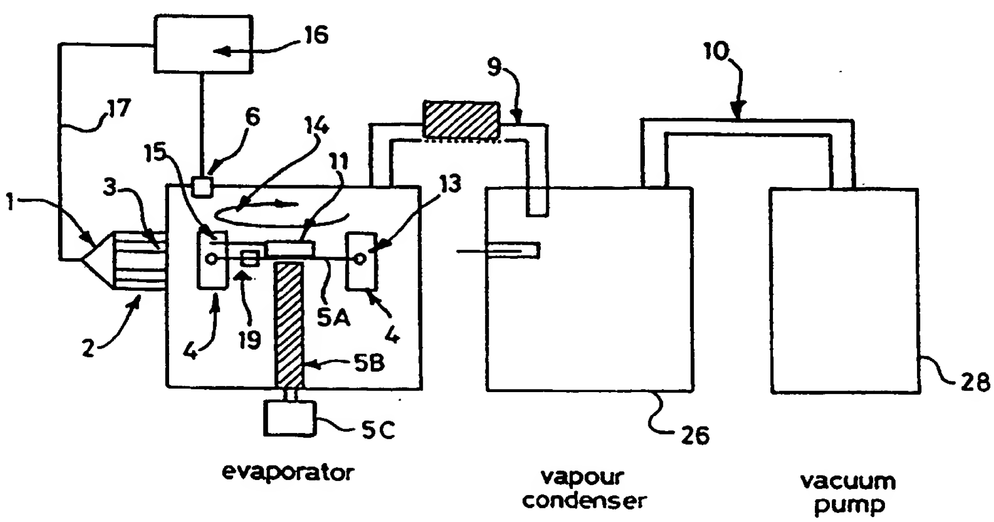
(57) **Abstract**



A method of controlling the evaporation of liquid in samples in an evaporating centrifuge, by monitoring the centrifugal force exerted on a sample holder containing a liquid sample having solid material dissolved or otherwise mixed therein. The centrifugal force is determined using a load cell (19), a strain gauge or, where relative movement between sample holder (4) and rotor (54) is permitted albeit with resilient restraining means, the centrifugal force signal may be generated by a position sensing transducer. The speed of rotation is sensed by a further transducer and both force and speed signals are conveyed to a computing means (54) programmed to generate a process control signal for controlling the evaporation process therefrom. A preferred method of control involves determining the rate of change of weight with time and terminating the evaporation process when the rate of change drops to zero. Evaporation is assisted by heating the samples and the process control signals determine not only the speed of rotation, but also the heating of the samples. A weight signal can be computed from the force signal by reference to the speed signal which is proportional to the centrifugal force acting on the sample holder and therefore the sample. A signal indicative of the weight of the sample itself can be computed by deducting from the combined weight of the holder and sample, a signal representing the weight of the sample holder itself. Different liquids evaporate at different rates and imbalance can occur as between different samples located around a rotor. Imbalance forces caused by differential evaporation rates may be reduced by means of a raceway (22) mounted on the rotor, or spindle driving rotor, which is incompletely filled with ball bearings (24) which in rotation distribute themselves around the raceway to counteract the imbalance forces.

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Title: Control of weight during evaporation of samples

Field of Invention

This invention relates to a method of and apparatus for controlling the weight of samples dissolved or suspended in a liquid while they are evaporating in a vacuum. It is particularly applicable to samples in centrifugal evaporators.

Background to the Invention

Samples to be evaporated in centrifugal evaporators are usually held in glass or plastic tubes or, sometimes, in a large number of small wells in plastic blocks. The sample holders are mounted upon a rotating assembly and spun at relatively high speed so that a considerable centrifugal force is applied to them in an outward direction, which forces the liquid to the lower part of the sample tubes and prevents any frothing or spitting of the liquid out of the sample tubes when a vacuum is applied. The spinning samples are held in a vacuum-tight chamber (referred to herein as a "chamber") which is connected to a vacuum pumping device.

Centrifugal evaporators of this type are well known and many types are available commercially. One problem from which such evaporators suffer, is that it is very difficult to obtain a desired continuous read-out of the weight of the sample in the holders as the liquid is being evaporated, since the holders are being spun at a high speed, typically at about 1400 r.p.m. The possibility has been considered of continuously weighing the whole evaporator during spinning. However, this involves measuring a total weight of the order of 50 kg to an accuracy of about 1 gm, which is a very demanding task.

Another problem arises when evaporation needs to take place simultaneously for different solvents, or solvent mixtures of differing compositions, in which the samples are dissolved

or suspended. In this situation those samples which are held in the more volatile solvents or mixtures will evaporate faster than the ones held in the less volatile solvents, and this can lead to an excessive imbalance in the rotating assembly, and consequent unwanted vibrations. This would also mitigate against the possibility of weighing the whole evaporator.

In most centrifugal evaporator machines such unwanted vibrations are arranged to trip an out-of-balance sensor to thereby stop the machine, but in machines without a sensor the vibrations can cause damage to the machine and even to the samples. Sometimes the vibration problem can be overcome by careful loading of the evaporator, or by stopping the process from time to time and rebalancing the load by adding liquid to empty samples or by rearranging the samples in the rotating assembly. Both these methods are tedious and time consuming.

It is an object of the present invention to enable the weight of a sample in a centrifugal evaporator to be continuously and accurately measured during evaporation.

It is another object of the invention to enable the operation of a centrifugal evaporator to continue despite a considerable imbalance of forces.

Summary of the invention

According to one aspect of the present invention, a method of evaporating a liquid sample contained in a sample holder which is mounted within a chamber and rotated by a rotor therein during the evaporation so that centrifugal force is exerted on the contents of the sample holder during the process whilst a pressure below atmospheric is maintained in the chamber in manner known per se, so as to leave as a residue any solid material dissolved or otherwise mixed in the liquid forming the sample, characterised by: mounting a transducer to monitor the force acting on the sample holder relative to the rotor when rotating at a given speed and obtaining a force signal therefrom, supplying the force signal to a computer

means, programming the computer means to compute a value equivalent to the centrifugal force exerted on the sample holder due to rotation of the rotor at said given speed, further programming the computer means to compute a weight value from the force signal using the computed centrifugal force, and further programming the computer means to generate a control signal for controlling the evaporation process in dependence on the computed weight value.

In some circumstances the rotor may be rotating at constant speed, so that the weight value can be computed for that particular speed.

Alternatively, however, the method may further comprise the steps of mounting a second transducer to monitor the speed of rotation of the rotor, obtaining a speed signal therefrom, and supplying the speed signal to the computing means for computing said weight value.

Preferably the computing means is adapted to rotate with the rotor.

Preferably the computing means is programmed to convert the output of the sensor into a form suitable for transmission to an external receiver.

Preferably the computing means converts the transducer signals into digital signals by which a carrier signal is modulated to effect the said transmission.

In general the transducer signals are produced continuously and the weight and centrifugal force factor values are continuously computed therefrom.

Conveniently the computing means has stored therein a value equivalent to the weight of the sample holder, and is further programmed to compute a value equivalent to the weight of the contents of the holder by deducting from the computed weight value a value equivalent to the known weight of the sample holder.

Preferably the computer means computes the rate of change of the computed weight value.

Preferably the method includes the step of heating the sample during rotation in the chamber to increase the rate of evaporation.

Preferably the method includes the step of controlling the supply of heat to the sample in dependence on the computed weight value, preferably in dependence on the computed rate of change of weight value.

In general, the supply of heat will be reduced as the rate of change of weight with time starts to decline, and the evaporation process is terminated when the rate of change drops to zero, indicating that the sample is dry.

The invention also lies in apparatus for evaporating a sample comprised of solid material dissolved or suspended in a liquid, comprising a vacuum chamber, a rotor therein, drive means for rotating the rotor relative to the chamber, a sample holder for containing the sample connected to the rotor, transducer means associated with the sample holder and the rotor for generating a force signal indicative of the centrifugal force acting on the sample holder as it is rotated at a given speed, and means for transmitting transducer signals to computing means programmed to convert the signal at any instant to a computer value proportional to weight, the computing means being further programmed to generate a process control signal for controlling the evaporation process in the chamber.

The force transducer may be a load cell, or a strain gauge, or where the sample holder is movable relative to the rotor, the force transducer may be a position sensor adapted to produce a signal indicating the position of the sample holder relative to the rotor, as determined by the instantaneous centrifugal force acting on the sample holder, causing it to move relative to the rotor.

Where the movement is permitted, preferably resilient means is provided which resists the movement of the sample holder relative to the rotor.

A plurality of sample holders may be mounted on the rotor and a force transducer is

provided for selected ones, or all of, the holders.

The weight of the sample can be calculated from a force value by taking account of the centrifugal force and deducting the known weight of the holder, but an equally useful measurement is that of the rate of change of weight. This is a direct measurement of mass flow rate and can be used to monitor the progress of the evaporation and to reduce the heat when the rate starts to decline, when the samples are nearly dry and to shut the system down when it drops to 0 indicating that the samples are dry.

According to another aspect of the invention in the processing of samples in a centrifugal evaporator in which the samples are dissolved or suspended in liquids of differing volatility, any imbalance caused during spinning of the rotor and resulting in unwanted vibration is at least partially compensated for by associating with the rotor an automatic balancing unit.

The invention therefore also lies in comprising a vacuum chamber, a rotor mounted therein for rotation in use about a generally vertical axis, a drive means for rotating the rotor, at least two sample holders mounted on the rotor, each sample holder being in use about a generally horizontal axis in a radial manner relative to the axis of rotation, a bearing raceway incorporating a plurality of ball bearings which do not fully occupy the circumferential extent of the raceway and which in rotation are automatically distributed around the raceway to counteract any imbalance forces experienced by the raceway, the bearing raceway being mounted to the rotor or a spindle driving the rotor, thereby to reduce any imbalance caused during the spinning of the rotor as result of differential evaporation of liquids from the sample holder.

The ball bearings may be formed from a high density material such as Tungsten or depleted Uranium.

The invention also lies in a method of measuring the weight of a liquid sample in a sample holder attached to a rotor in a vacuum chamber of an evaporating centrifuge, comprising the steps of mounting a transducer to monitor the force acting on the sample holder relative to

the rotor during rotation, supplying a force signal to a computing means having stored therein a stored weight value corresponding to the empty weight of the sample holder, the computing means being programmed to convert the force signal to a weight value for a given speed of rotation of the rotor, the computing means being further programmed to deduct from the computed weight value said stored weight value.

The method may further comprise the steps of monitoring the speed of rotation of the rotor, and supplying a speed signal to the computing means for computing said weight signal.

The weight measuring method may be enhanced by mounting to the rotating parts of the apparatus an automatic balancing aid, to counteract any out of balance force arising from differential evaporation of samples.

Only limited space is available within apparatus as described herein for laboratory use and the like, and therefore it is to advantage to use rolling elements constructed from dense materials such as Tungsten or depleted Uranium, since this allows the overall size of the raceway to be reduced both in depth and diameter, due to the increased mass of the rolling elements obtained by using high density materials therefore.

Brief description of the drawings

The invention will now be described, by way of example only, with reference to the accompanying drawings in which:

Figure 1 is a schematic side view of a centrifugal evaporation system incorporating a force measuring transducer in accordance with the invention;

Figure 2 is a perspective view of a dissembled automatic balancing unit associated with the rotor of Figure 1, and

Figures 3 and 4 are block schematic diagrams showing the probes and control system as

employed in an evaporator such as shown in Figure 1 embodying the invention.

Detailed description

Figure 1 illustrates a centrifugal evaporator embodying the invention described and claimed herein.

The samples in Figure 1 are contained in plates or blocks 4 in which there are numerous sample wells (not shown), commonly referred to as deep-well microtitre plates or blocks.

When the sample holder rotor 5A and shaft 5B rotates, driven by a motor 5C, which may be inside but more usually external to the chamber (14), the sample blocks swing out to a position in which the sample wells are horizontal, under the influence of centrifugal force.

The sample blocks are pivoted about swivel pins 13 and the blocks are held with the wells vertical for loading in the a stationary evaporator. Vacuum is then applied to the evaporator chamber 14 via pipe 9 from a vapour condenser 26 which in turn is pumped via pipe 10 by a vacuum pump 28.

Heat is applied to the rotating sample blocks 4 by a heater 1 in the form of a high temperature infra-red radiation source, and a beam of radiant heat energy 2 passes through a window 3 of heat-transparent material such as quartz which is sealed into the wall of the vacuum chamber 14 and reaches the sample holder as illustrated.

A temperature sensor or probe 15 is placed in one of the sample wells, or otherwise placed in close proximity to the wells in one of the sample blocks, and is connected to a transmitter 11 which transmits signals corresponding to the sample temperature to an aerial and feedthrough 6 inside and extending through the chamber wall, and which is connected to an external receiver and decoder 16. The decoder includes data processing and computing facilities, as required, and indicates the sample temperature by a display (not shown) and, if required, can be programmed to generate electrical signals to control the operation of the

heater in order to increase or decrease the heat energy to keep the samples at desired temperatures during the process. Such control signals are supplied to the heater 1 via a connection 17.

It is important that as far as possible all the samples evaporate at the same rate. To achieve this, all the samples should receive the same heat input by directing the heat to them so as to heat all the sample holders uniformly. A common form of sample holder is the deep-well microtitre plate or block 4, in which there are typically 96 wells.

Each block 4 is mounted on the swivel pin 13 so that when it is initially loaded onto a stationary rotor 5A the open ends of the wells face upwards; but as soon as the rotor 5A is rotated at a sufficient speed, the blocks 4 swing into a position in which the wells are almost horizontal, as is in fact shown in Figure 1. In this position the infra-red beam 2 is directed horizontally onto the closed ends of the sample wells, in which configuration it is possible to achieve uniform heating of the wells.

Even with perfectly uniform heat input the samples will not evaporate at a uniform rate because of a so-called "cold neighbour effect". If the samples are in thermal contact with each other, as is the case for example in a microtitre plate or block 4, the outer samples only have evaporating (and therefore "cold") neighbours on three or (for corner samples) two sides, and therefore do not lose as much heat to their neighbours as those in the centre which have four "cold" neighbours. Also two of an outside sample's neighbours will generally be less cold than those of the inner samples. Outer samples therefore can evaporate faster than centrally located samples.

This effect can be reduced or eliminated by reducing the heat input to the outer samples. A simple way of doing this in the preferred infra-red heating case, is to provide graduated shading from the infra-red beam 2 by, for example, placing a metal screen between the sample holder and the heater 1. The screen contains graduated perforations so that those in the outer region transmit much less radiation than do those in the central region, and those in intermediate regions, which have an intermediate size thereby transmit greater quantities

of heat than do the outer ones.

Although the sample holder (4) illustrated is described as being a deep-well microtitre block or plate, the same techniques may be employed to obtain uniform temperature and graduated heating as described above, when using arrays of tubes, bottles or vials in holders which swing out on swivels in a similar manner.

The power of the heater 1 is controlled by measuring sample temperature or chamber pressure and taking appropriate steps to raise or lower the heater power. Thus at the start of the process a high heat input is required, but as the samples approach dryness the evaporation rate will reduce and the sample temperature will start to rise so that the heat input must be reduced to avoid overheating the sample, and when the samples are dry, the heating must be discontinued.

The vapour condenser 26 is used in centrifugal evaporation equipment to increase pumping speed for the liquid being evaporated and to protect the vacuum pump 28 from vapours which might impair its efficiency. Such a condenser is a vessel held at low temperatures at which the vapours being evaporated condense or solidify.

If the condenser 26 is located between the vacuum pump 28 and the evaporation chamber 14, as shown in Figure 1, the pressure in the chamber 14 cannot be reduced below the vapour pressure of any condensed liquid remaining in the condenser 26. This is due to the evaporation of condensed material which will take place in the condenser if the system pressure is reduced to a level approaching the vapour pressure of the condensed material left in the condenser 26. This phenomenon, especially if a more volatile material has been left in the condenser 26 from a previous run, can make chamber pressure a rather insensitive technique for sensing sample temperature at the end of evaporation to indicate when the samples are dry, and it may be unreliable as a means for determining when the equipment can be shut down.

The measurement of vapour flow rate is a more useful monitor of the evaporation process.

By thus monitoring flow rate, information can be obtained about a process to indicate when to turn off the heater, since when the samples are nearly dry the flow rate will become low. This enables equipment to be reliably shut down when the process is finished (ie the samples are dry).

Flow rate through the condenser or the pipe 9 between the chamber 14 and the condenser 26 can be monitored by any convenient technique.

In accordance with the present invention, a load cell 19 is attached between each plate or block 4 and its support. The load cell produces an electrical signal indicative of the horizontal force on the block which, when the rotor is spinning, will be proportional to the combined weight of the sample and the sample holding assembly. Since the latter is constant the sample weight can readily be obtained. Of course, the apparent weight will be exaggerated by a factor due to the centrifugal force, but this factor will not vary for a given rotor speed. In some arrangements the rotor speed may be kept constant; however, where the speed is variable it is important also to monitor the rotational speed of the rotor and sample holders.

Figure 3 shows the important components of the monitoring system for a chamber 14, such as shown in Figure 1. Each temperature probe 15 connects to an input of a signal processor 50, the output of which is digitised by an A/D converter 52 for supply to a microprocessor 54 which handles the modulation of a radio signal in a transmitter 56 to which signals are supplied from the microprocessor for radiation by an antenna 58. Power for the system may be from a battery or a mains supply 60. Except for the probe 15 and the antenna 58, all the units shown in Figure 3 may be housed within a housing located on the sample holder rotor 5A, so that there is no relative movement between the housing and the probe 15. The chamber 14 must be constructed so that at least part of its wall is capable of transmitting the radio signals from the antenna.

The force signals from the load cell 19 are processed and transmitted to a receiver and decoder outside the chamber via a separate transmission channel on the signal processing

circuit of Figure 3.

A receiver and control system for locating outside the chamber 14 is shown in Figure 4.

Here the receiver antenna 62 feeds radio signals to a receiver and decoder 64 which supplies decoded digital data signals (corresponding to those from the A/D converter 52 in Figure 3), to a second microprocessor 66. This controls the supply of digital signals to a motor controller 68 which controls the speed of rotation of the drive motor 5C (also shown in Figure 1). A tacho-generator 70 is attached to the motor shaft 72 and provides a speed signal for the microprocessor 66.

An infra-red heater 1 (see also Figure 1) is controlled by a power controller 74 which in turn is controlled by signals from the microprocessor 66, to reduce the heat output from the heater 1 as an evaporation process progresses, so as to reduce the risk of overheating as samples dry and are no longer cooled by evaporative cooling effects.

The vacuum pump 28 of Figure 1 is shown associated to the chamber 14 via a pipeline 76 which includes a valve 78 also under the control of signals from the microprocessor 66. The latter includes a memory in which operating system software and data relating to different volatile liquids are stored and a data entry keyboard or other device 80 allows data to be entered initially and volatile components to be identified to the system. A display screen 82 assists in the entry of data and the display of monitored values of temperature from probe 15 and pressure from a probe 84 in the chamber, and of force (and therefore by computation weight) from load cell 19.

The memory also stores the values of force signals from load cell 19 when an empty standard sample holder is rotating around the chamber, and factors by which force signal values can be converted to weight for different rotational speeds (from the tacho 70) and therefore different g-forces. It can also store weight values for empty sample holders such as mitrotitre plates or blocks.

Power for the system of Figure 4 may be from a battery or a mains driven power supply 86.

Experiments have shown that weights of samples in holders weighing up to 1200 gm can be determined using this apparatus and approved to an accuracy of better than 1 gm.

The microprocessor 66 can be programmed to compute the rate of change of weight with time, and this or the monitored force value can be used to determine when the samples have been fully evaporated, and therefore the point at which the samples are completely dry. This enables the correct moment to be identified when to switch off heat to the samples.

Figure 2 shows a proprietary automatic balancing unit 20,22 which is fitted to the rotor shaft 5B as close as possible to the rotor 5A carrying the plates or blocks 4. Vibration caused by rotor imbalance is likely to occur when solvents of different volatility are used for the samples.

The unit 20, 22 may be an Auto-Balancing unit produced by the bearing manufacturing company SKF.

As shown in Figure 2, the unit comprises inner and outer raceways 20 and 22 between which a number of loose ball bearings 24 are freely movable. The ball bearings distribute themselves automatically to counteract the imbalance in the rotor shaft 5.

In the known forms of autobalancing units of this type the ball bearings 24 are normally made of steel, but a greater balancing capability can be obtained by using balls of a heavier material, for example Tungsten or depleted Uranium. The use of higher density metal for the balls allows the same out-of-balance forces to be counteracted using a raceway assembly 20, 22 of small dimensions, both in width and diameter.

One unit which has been used to advantage is the Auto-Balancing device produced by the company SKF such as is described in WO98/01733.

CLAIMS

1. A method of evaporating a liquid sample contained in a sample holder which is mounted within a chamber and rotated by a rotor therein during the evaporation so that centrifugal force is exerted on the contents of the sample holder during the process whilst a pressure below atmospheric is maintained in the chamber in manner known per se, so as to leave as a residue any solid material dissolved or otherwise mixed in the liquid forming the sample, characterised by:

mounting a transducer to monitor the force acting on the sample holder relative to the rotor when rotating at a given speed and obtaining a force signal therefrom, supplying the force signal to a computer means, programming the computer means to compute a value equivalent to the centrifugal force exerted on the sample holder due to rotation of the rotor at said given speed, further programming the computer means to compute a weight value from the force signal using the computed centrifugal force, and further programming the computer means to generate a control signal for controlling the evaporation process in dependence on the computed weight value.

2. A method as claimed in claim 1, further comprising the steps of mounting a second transducer to monitor the speed of rotation of the rotor, obtaining a speed signal therefrom, and supplying the speed signal to the computing means for computing said weight value.

3. A method as claimed in claim 1 or claim 2, wherein the computing means is adapted to rotate with the rotor.

4. A method as claimed in any one preceding claim, wherein the computing means is programmed to convert the transducer signals into a form suitable for transmission to an external receiver.

5. A method as claimed in claim 4, wherein the computing means converts the transducer signals into digital signals by which a carrier signal is modulated to effect the said transmission.

6. A method as claimed in any one of claims 1 to 5, wherein the force and speed signals are produced continuously and the weight and centrifugal force values are continuously computed therefrom.

7. A method as claimed in claim 6, wherein the computing means has stored therein a value equivalent to the weight of the sample holder, and is further programmed to compute a value equivalent to the weight of the contents of the holder by deducting from the computed weight value a value equivalent to the known weight of the sample holder.

8. A method as claimed in any one preceding claim, wherein the computer means computes the rate of change of the computed weight value.

9. A method as claimed in any one preceding claim, further comprising the step of heating the sample during rotation in the chamber to increase the rate of evaporation.

10. A method as claimed in claim 9, comprising the step of controlling the supply of heat to the sample in dependence on the computed weight value.

11. A method as claimed in claim 8, comprising the step of controlling the supply of heat in dependence on the computed rate of change of weight value.

12. A method as claimed in claim 11, wherein the supply of heat is reduced as the rate of change of weight with time starts to decline, and the evaporation process is terminated when the rate of change drops to zero, indicating that the sample is dry.

13. Apparatus for evaporating a sample comprised of solid material dissolved or suspended in a liquid, comprising a vacuum chamber, a rotor therein, drive means for rotating the rotor relative to the chamber, a sample holder for containing the sample

connected to the rotor, transducer means associated with the sample holder and the rotor for generating a force signal indicative of the centrifugal force acting on the sample holder as it is rotated at a given speed, and means for transmitting transducer signals to computing means programmed to convert the signal at any instant to a computed value proportional to weight, the computing means being further programmed to generate a process control signal for controlling the evaporation process in the chamber.

14. Apparatus as claimed in claim 13, further comprising second transducer means associated with the rotor for generating a speed signal corresponding to the speed of rotation of the rotor, the speed signal being transmitted to the computing means for computing a weight value.

15. Apparatus as claimed in claim 13 or claim 14, wherein the first mentioned transducer is a load cell.

16. Apparatus as claimed in claim 13 or claim 14, wherein the first mentioned transducer is a strain gauge.

17. Apparatus as claimed in claim 13 or claim 14, wherein the sample holder is movable relative to the rotor and the force transducer is a position sensor adapted to produce a signal indicating the position of the sample holder relative to the rotor, as determined by the instantaneous centrifugal force acting on the sample holder, causing it to move relative to the rotor.

18. Apparatus as claimed in claim 17 wherein resilient means resists the movement of the sample holder relative to the rotor.

19. Apparatus as claimed in any one of claims 13 to 18, wherein a plurality of sample holders are mounted on the rotor and a force transducer is provided for at least selected ones the holders.

20. Apparatus as claimed in any one of claims 13 to 19, wherein a mechanical device is

attached to the rotor or a spindle on which the rotor is carried and by which it is rotated, which automatically adjusts its centre of mass in response to out-of-balance forces acting on the rotor due to differential evaporation of samples.

21. Apparatus comprising a vacuum chamber, a rotor mounted therein for rotation in use about a generally vertical axis, a drive means for rotating the rotor, at least two sample holders mounted on the rotor, each sample holder being pivotal in use about a generally horizontal axis in a radial manner relative to the axis of rotation, a bearing raceway incorporating a plurality of ball bearings which do not fully occupy the circumferential extent of the raceway and which in rotation are automatically distributed around the raceway to counteract any imbalance forces, the raceway being mounted to the rotor or a spindle driving the rotor, thereby to reduce any imbalance caused during the spinning of the rotor as result of differential evaporation of liquids from the sample holder.

22. Apparatus as claimed in claim 21, wherein the ball bearings are formed from a high density material such as Tungsten or depleted Uranium.

23. A method of measuring the weight of a liquid sample in a sample holder attached to a rotor in a vacuum chamber of an evaporating centrifuge, comprising the steps of mounting a transducer to monitor the force acting on the sample holder relative to the rotor during rotation, supplying a force signal to a computing means having stored therein a stored weight value corresponding to the empty weight of the sample holder, the computing means being programmed to convert the force signal to a weight value for a given speed of rotation of the rotor, the computing means being further programmed to deduct from the computed weight value said stored weight value.

24. A method as claimed in claim 23, further comprising the steps of monitoring the speed of rotation of the rotor, and supplying a speed signal to the computing means for computing said weight signal.

25. Centrifugal evaporation means including weight monitoring and process control means constructed and arranged substantially as herein described with reference to and as

illustrated in the accompanying drawings.

26. Methods of monitoring weight and rate of change of weight of evaporating samples, and of controlling a centrifugal evaporation process in dependence on the monitored weight or rate of change of weight, substantially as herein described with reference to and as illustrated in the accompanying drawings.

09/508133

1/4

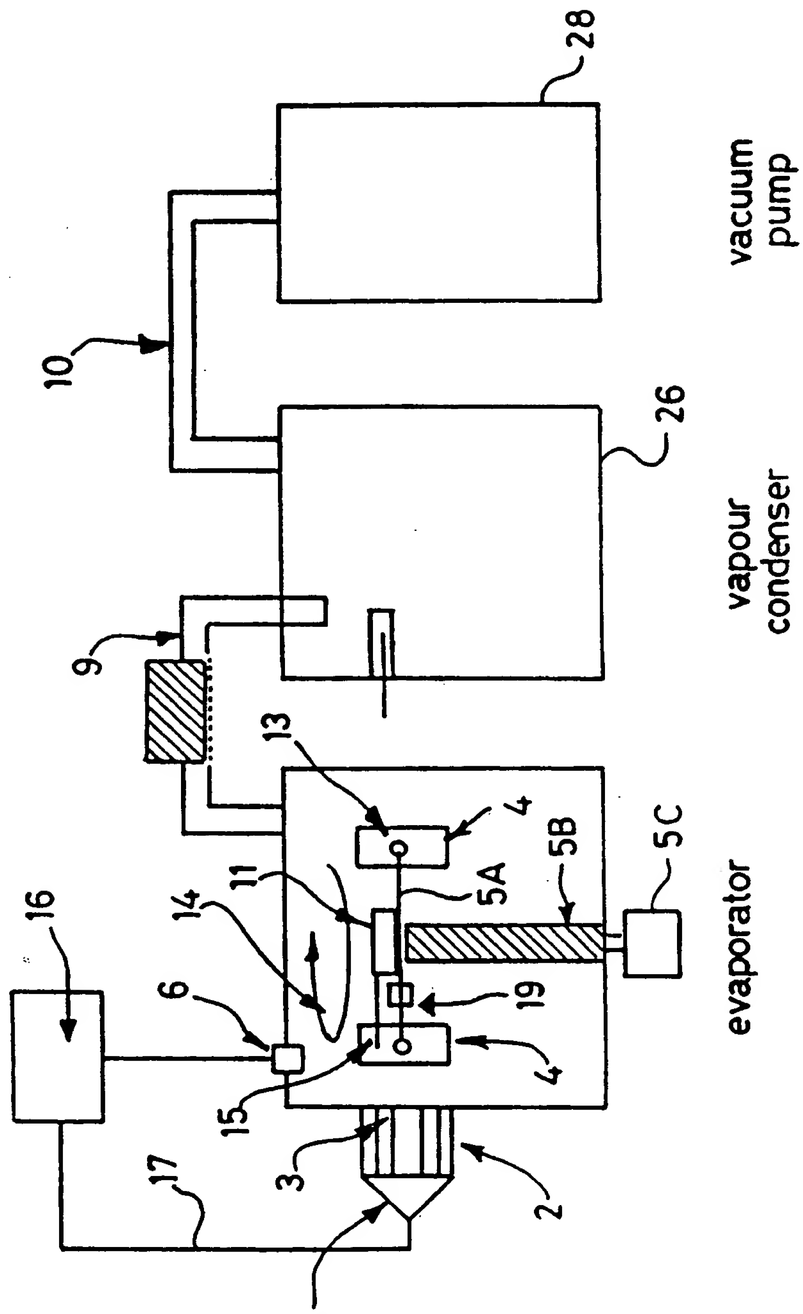


Fig. 1

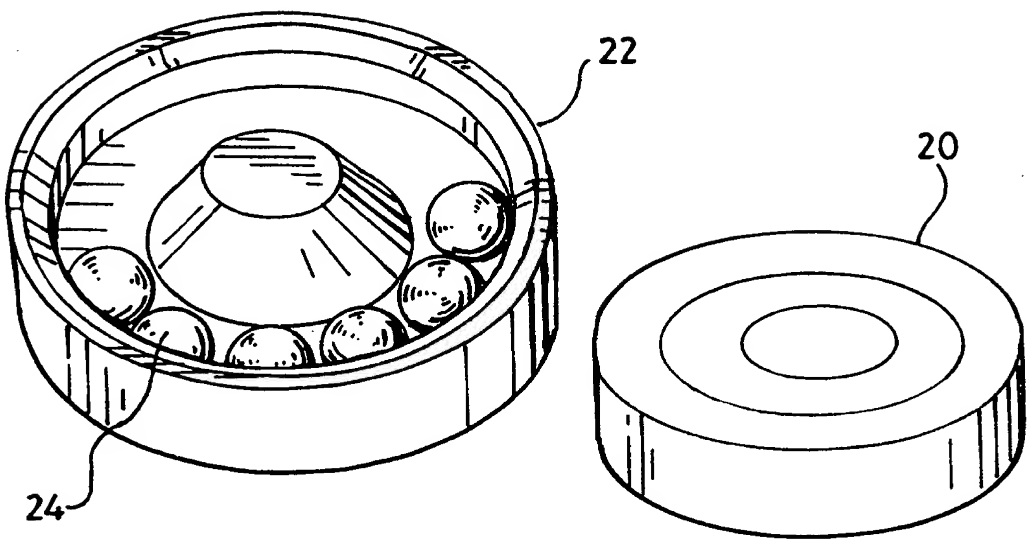


Fig. 2

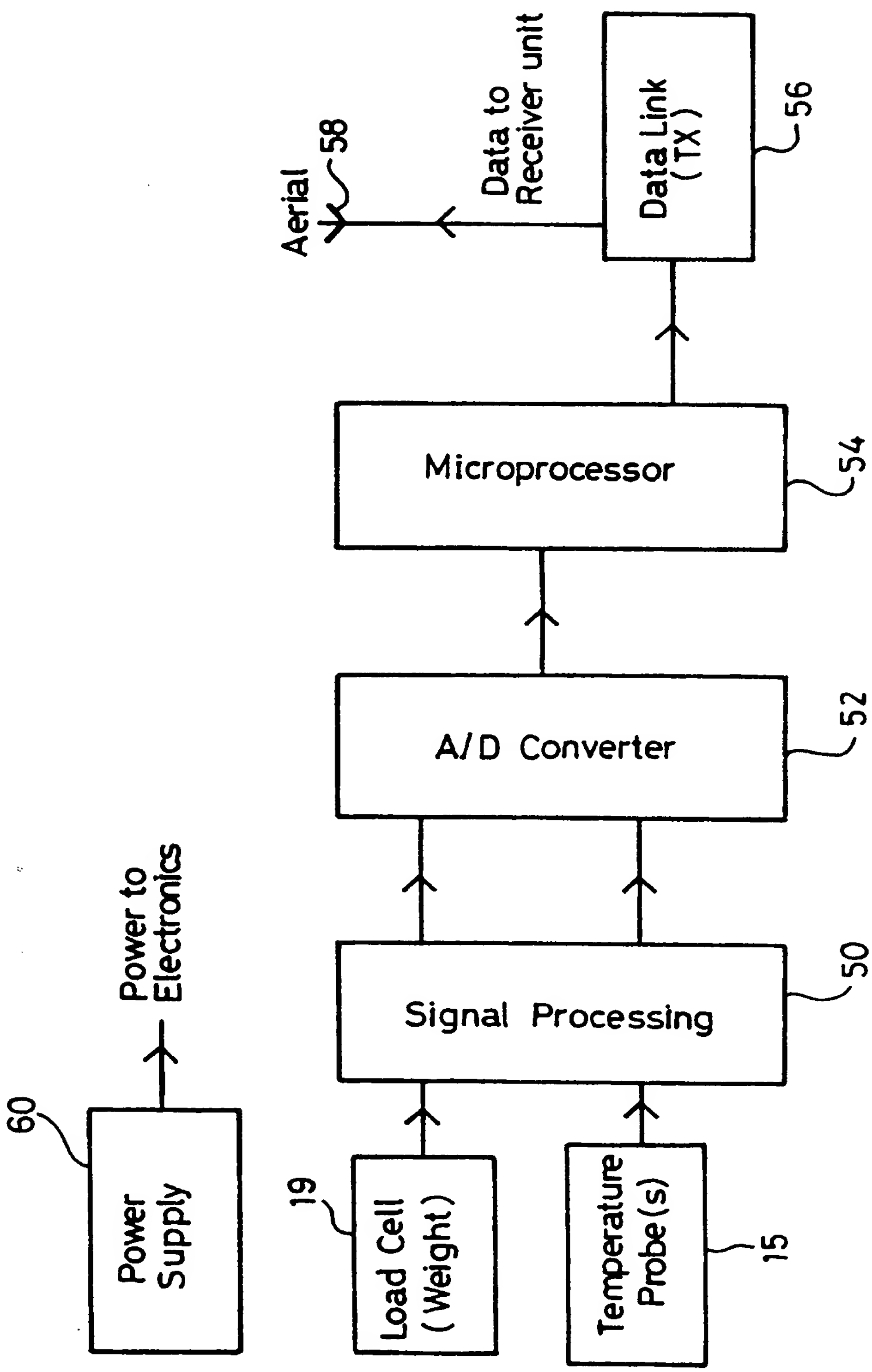

Aerial
58
Data to Receiver unit
Data Link (TX)
56
Microprocessor
54
A/D Converter
52
Power to Electronics
Signal Processing
50
60
Power Supply
19
Load Cell (Weight)
Temperature Probe(s)
15

Fig. 3

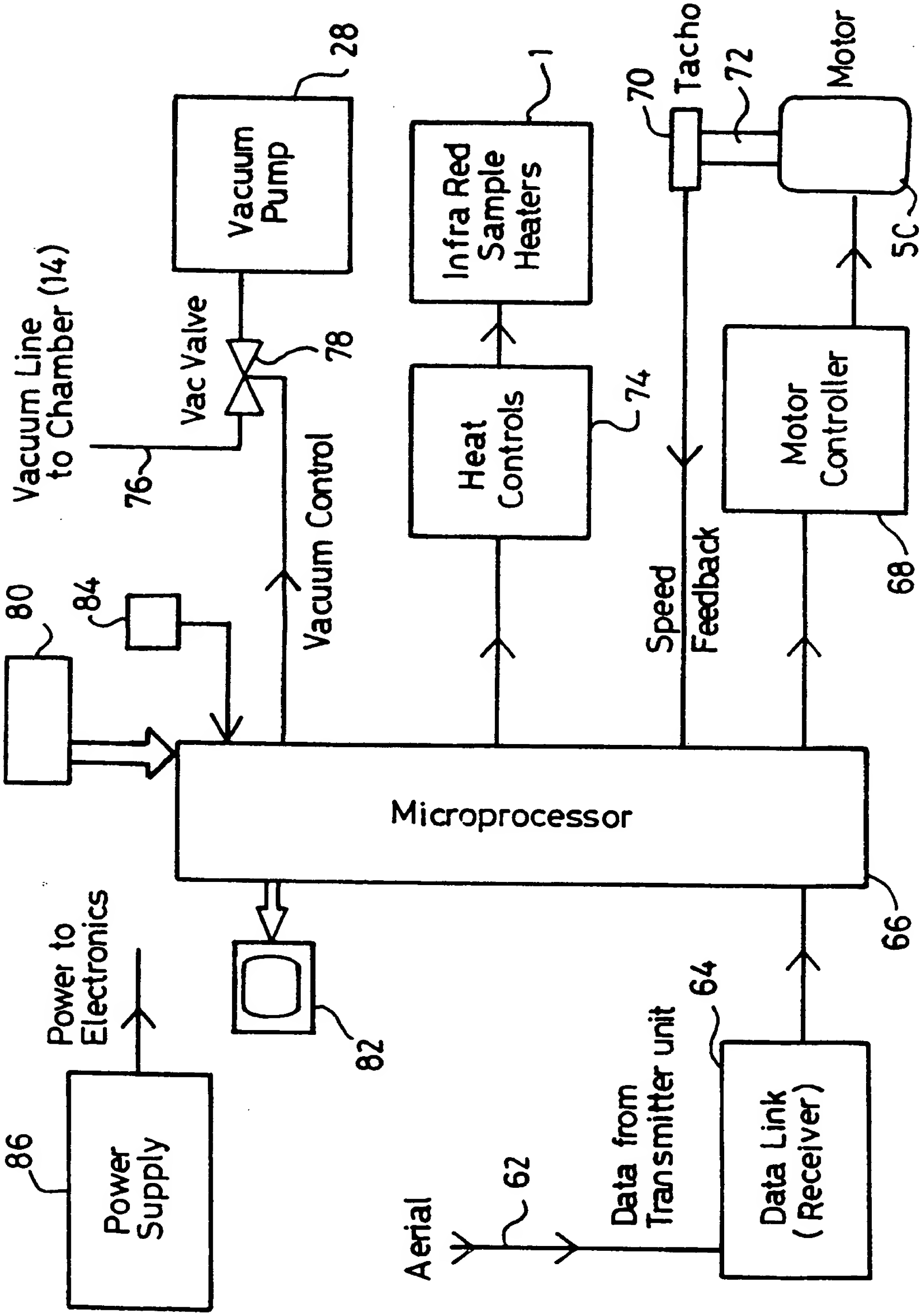

Vacuum Pump
28
Infra Red Sample Heaters
1
70
Tacho
72
Motor
5C
Vacuum Line to Chamber (14)
Vac Valve
78
76
Vacuum Control
Heat Controls
74
Speed Feedback
Motor Controller
68
80
84
Microprocessor
66
Power to Electronics
82
Power Supply
86
Aerial
62
Data from Transmitter unit
64
Data Link (Receiver)

Fig. 4

5640
09/508133
1764

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 7 : G01G 9/00, B01D 3/42, 3/08, B04B 13/00, G01N 1/40, 5/04	A3	(11) International Publication Number: WO 00/14493 (43) International Publication Date: 16 March 2000 (16.03.00)

(21) International Application Number: PCT/GB99/01790

(22) International Filing Date: 7 June 1999 (07.06.99)

(30) Priority Data:
9819286.7 5 September 1998 (05.09.98) GB
9824703.4 12 November 1998 (12.11.98) GB

(71)(72) Applicant and Inventor: COLE, Michael [GB/GB]; Poplar Farm, Low Road, Marlesford, Woodbridge, Suffolk IP13 0AL (GB).

(74) Agent: KEITH W NASH & CO.; 90–92 Regent Street, Cambridge CB2 1DP (GB).

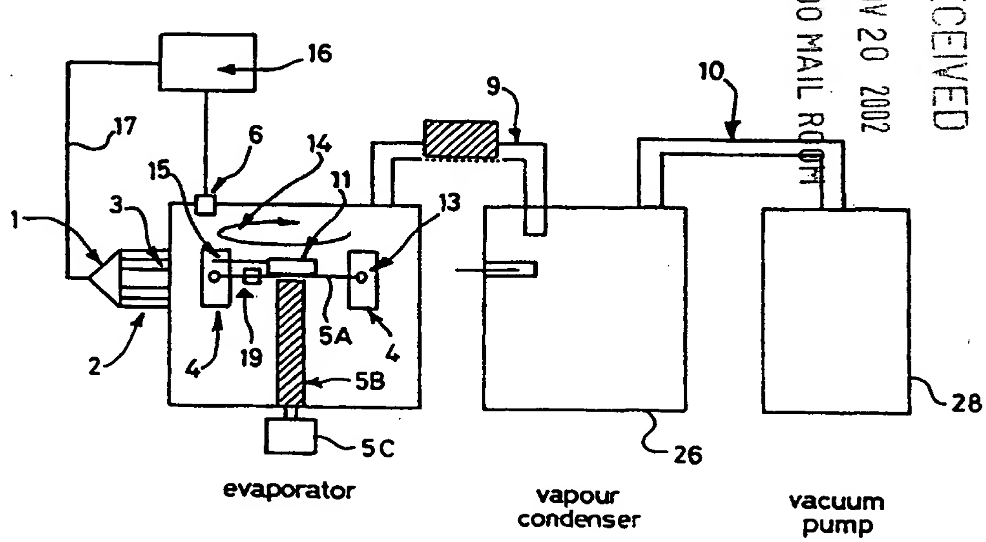
(81) Designated States: JP, US, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

Published
With international search report.

(88) Date of publication of the international search report: 8 June 2000 (08.06.00)

(54) Title: CONTROL OF WEIGHT DURING EVAPORATION OF SAMPLES

(57) Abstract



A method of controlling the evaporation of liquid in samples in an evaporating centrifuge, by monitoring the centrifugal force exerted on a sample holder containing a liquid sample having solid material dissolved or otherwise mixed therein. The centrifugal force is determined using a load cell (19), a strain gauge or, where relative movement between sample holder (4) and rotor (54) is permitted albeit with resilient restraining means, the centrifugal force signal may be generated by a position sensing transducer. The speed of rotation is sensed by a further transducer and both force and speed signals are conveyed to a computing means (54) programmed to generate a process control signal for controlling the evaporation process therefrom. A preferred method of control involves determining the rate of change of weight with time and terminating the evaporation process when the rate of change drops to zero. Evaporation is assisted by heating the samples and the process control signals determine not only the speed of rotation, but also the heating of the samples. A weight signal can be computed from the force signal by reference to the speed signal which is proportional to the centrifugal force acting on the sample holder and therefore the sample. A signal indicative of the weight of the sample itself can be computed by deducting from the combined weight of the holder and sample, a signal representing the weight of the sample holder itself. Different liquids evaporate at different rates and imbalance can occur as between different samples located around a rotor. Imbalance forces caused by differential evaporation rates may be reduced by means of a raceway (22) mounted on the rotor, or spindle driving rotor, which is incompletely filled with ball bearings (24) which in rotation distribute themselves around the raceway to counteract the imbalance forces.

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BA	Bosnia and Herzegovina	**GE**	Georgia	**MD**	Republic of Moldova	**TG**	Togo
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CI	Côte d'Ivoire	**KP**	Democratic People's Republic of Korea	**NZ**	New Zealand		
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CN	China	**KR**	Republic of Korea	**PT**	Portugal		
CU	Cuba	**KZ**	Kazakstan	**RO**	Romania		
CZ	Czech Republic	**LC**	Saint Lucia	**RU**	Russian Federation		
DE	Germany	**LI**	Liechtenstein	**SD**	Sudan		
DK	Denmark	**LK**	Sri Lanka	**SE**	Sweden		
EE	Estonia	**LR**	Liberia	**SG**	Singapore		

INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 99/01790

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 G01G9/00 B01D3/42 B01D3/08 B04B13/00 G01N1/40
G01N5/04

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 G01G B01D B04B G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	DE 195 18 540 A (LAUTSCHLAEGER WERNER) 21 November 1996 (1996-11-21) column 7, line 26 -column 8, line 33; figure 1	1,13,23
Y	EP 0 128 590 A (RESEARCH CORP) 19 December 1984 (1984-12-19) page 48, line 15 -page 49, line 15; figure 21	1,13,23
A	US 4 157 781 A (MARUYAMA HITOSHI) 12 June 1979 (1979-06-12) abstract; figure 1	1,13,23
A	DE 42 11 760 C (ERNO RAUMFAHRTTECHNIK GMBH) 19 August 1993 (1993-08-19) abstract	1,13,23

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
30 August 1999	09.03.2000

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	GANCI P.A.

1

INTERNATIONAL SEARCH REPORT

International application No.
PCT/GB 99/01790

Box I Observati ns wh r certain claims w r found uns archabl (Continuation of item 1 of first sh et)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. ☒ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:

   1-20,23,34

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest.

☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

International Application No. PCT/GB 99/01790

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

1. Claims: 1-20,23,24

   Control of weight during evaporation of sample in centrifugal evaporators

2. Claims: 21,22

   Device for reducing imbalancing in a centrifuge.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/GB 99/01790

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
DE 19518540 A	21-11-1996	NONE	
EP 0128590 A	19-12-1984	CA 1232153 A	02-02-1988
		DE 3473511 A	22-09-1988
		IL 72090 A	31-10-1988
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		US 4589782 A	20-05-1986
		US 4596470 A	24-06-1986
US 4157781 A	12-06-1979	NONE	
DE 4211760 C	19-08-1993	NONE	

International Application No
PCT/GB 99/01790

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 692 236 A (LIVSHITZ STANLEY L ET AL) 19 September 1972 (1972-09-19) column 4, line 11 -column 5, line 19; figure 2	21,22
X	DE 197 49 357 A (HITACHI KOKI KK) 25 June 1998 (1998-06-25) column 1, line 17 - line 28; figure 21	21,22

2

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/GB 99/01790

Patent document cited in search report		Publication date	Patent family member(s)			Publication date
DE 19518540	A	21-11-1996	NONE			
EP 0128590	A	19-12-1984	CA	1232153	A	02-02-1988
			DE	3473511	D	22-09-1988
			IL	72090	A	31-10-1988
			JP	60057243	A	03-04-1985
			US	4589782	A	20-05-1986
			US	4596470	A	24-06-1986
US 4157781	A	12-06-1979	NONE			
DE 4211760	C	19-08-1993	NONE			
US 3692236	A	19-09-1972	AU	3333171	A	15-03-1973
			BE	773364	A	30-03-1972
			CA	936506	A	06-11-1973
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			FR	2110116	A	26-05-1972
			GB	1346115	A	06-02-1974
			IT	938889	B	10-02-1973
			NL	7114176	A	03-05-1972
			SU	463254	A	05-03-1975
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REVISED VERSION*

QV 89508194 1764

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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 7 : **G01G 9/00, B01D 3/42, 3/08, B04B 13/00, G01N 1/40, 5/04, B04B 9/14** — A3

(11) International Publication Number: **WO 00/14493**

(43) International Publication Date: 16 March 2000 (16.03.00)

(21) International Application Number: PCT/GB99/01790

(22) International Filing Date: 7 June 1999 (07.06.99)

(30) Priority Data:
9819286.7 5 September 1998 (05.09.98) GB
9824703.4 12 November 1998 (12.11.98) GB

(71)(72) Applicant and Inventor: COLE, Michael [GB/GB]; Poplar Farm, Low Road, Marlesford, Woodbridge, Suffolk IP13 0AL (GB).

(74) Agent: KEITH W NASH & CO.; 90–92 Regent Street, Cambridge CB2 1DP (GB).

(81) Designated States: JP, US, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

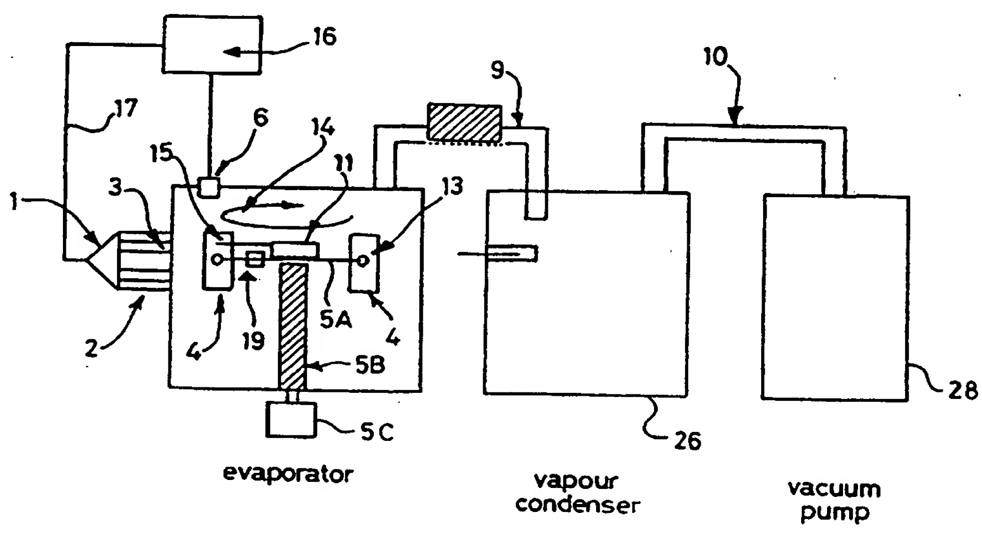
Published
With a revised version of the international search report.

(88) Date of publication of the international search report: 8 June 2000 (08.06.00)

(88) Date of publication of the revised version of the international search report: 10 August 2000 (10.08.00)

(54) Title: CONTROL OF WEIGHT DURING EVAPORATION OF SAMPLES

(57) Abstract



A method of controlling the evaporation of liquid in samples in an evaporating centrifuge, by monitoring the centrifugal force exerted on a sample holder containing a liquid sample having solid material dissolved or otherwise mixed therein. The centrifugal force is determined using a load cell (19), a strain gauge or, where relative movement between sample holder (4) and rotor (54) is permitted albeit with resilient restraining means, the centrifugal force signal may be generated by a position sensing transducer. The speed of rotation is sensed by a further transducer and both force and speed signals are conveyed to a computing means (54) programmed to generate a process control signal for controlling the evaporation process therefrom. A preferred method of control involves determining the rate of change of weight with time and terminating the evaporation process when the rate of change drops to zero. Evaporation is assisted by heating the samples and the process control signals determine not only the speed of rotation, but also the heating of the samples. A weight signal can be computed from the force signal by reference to the speed signal which is proportional to the centrifugal force acting on the sample holder and therefore the sample. A signal indicative of the weight of the sample itself can be computed by deducting from the combined weight of the holder and sample, a signal representing the weight of the sample holder itself. Different liquids evaporate at different rates and imbalance can occur as between different samples located around a rotor. Imbalance forces caused by differential evaporation rates may be reduced by means of a raceway (22) mounted on the rotor, or spindle driving rotor, which is incompletely filled with ball bearings (24) which in rotation distribute themselves around the raceway to counteract the imbalance forces.

*(Referred to in PCT Gazette No. 32/2000, Section II)

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GA	Gabon
GB	United Kingdom
GE	Georgia
GH	Ghana
GN	Guinea
GR	Greece
HU	Hungary
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IS	Iceland
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JP	Japan
KE	Kenya
KG	Kyrgyzstan
KP	Democratic People's Republic of Korea
KR	Republic of Korea
KZ	Kazakstan
LC	Saint Lucia
LI	Liechtenstein
LK	Sri Lanka
LR	Liberia
LS	Lesotho
LT	Lithuania
LU	Luxembourg
LV	Latvia
MC	Monaco
MD	Republic of Moldova
MG	Madagascar
MK	The former Yugoslav Republic of Macedonia
ML	Mali
MN	Mongolia
MR	Mauritania
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VN	Viet Nam
YU	Yugoslavia
ZW	Zimbabwe

REVISED VERSION

INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 99/01790

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 G01G9/00 B01D3/42 B01D3/08 B04B13/00 G01N1/40
G01N5/04 B04B9/14

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 G01G B01D B04B G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	DE 195 18 540 A (LAUTSCHLAEGER WERNER) 21 November 1996 (1996-11-21) column 7, line 26 -column 8, line 33; figure 1 ---	1,13,23
Y	EP 0 128 590 A (RESEARCH CORP) 19 December 1984 (1984-12-19) page 48, line 15 -page 49, line 15; figure 21 ---	1,13,23
A	US 4 157 781 A (MARUYAMA HITOSHI) 12 June 1979 (1979-06-12) abstract; figure 1 ---	1,13,23
A	DE 42 11 760 C (ERNO RAUMFAHRTTECHNIK GMBH) 19 August 1993 (1993-08-19) abstract ---	1,13,23
	-/--	

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
28 April 2000	09.06.00

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Ganci, P

2

International Application No
PCT/GB 99/01790

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 692 236 A (LIVSHITZ STANLEY L ET AL) 19 September 1972 (1972-09-19) column 4, line 11 -column 5, line 19; figure 2	21,22
X	DE 197 49 357 A (HITACHI KOKI KK) 25 June 1998 (1998-06-25) column 1, line 17 - line 28; figure 21	21,22

2

International application No.
PCT/GB 99/01790

INTERNATIONAL SEARCH REPORT

B x I Observati ns where certain claims wer found unsearchabl (C ntinuation of item 1 f first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons.

1. [ ] Claims Nos :
because they relate to subject matter not required to be searched by this Authority, namely

2. [X] Claims Nos. 25,26
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:
Claims 25 and 26 are unacceptable according to PCT Rule 6.2(a) and PCT Article 6

3. [ ] Claims Nos.
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1. [X] As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.

2. [ ] As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. [ ] As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:

4. [ ] No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims, it is covered by claims Nos.:

Remark on Protest

[ ] The additional search fees were accompanied by the applicant's protest.

[X] No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box I.2

Claims Nos.: 25,26

Claims 25 and 26 are unacceptable according to PCT Rule 6.2(a) and PCT Article 6

The applicant's attention is drawn to the fact that claims, or parts of claims, relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/GB 99/01790

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
DE 19518540 A	21-11-1996	NONE	
EP 0128590 A	19-12-1984	CA 1232153 A	02-02-1988
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PATENT COOPERATION TREATY

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PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference C1071.01/C	**FOR FURTHER ACTION** See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/GB99/01790	International filing date *(day/month/year)* 07/06/1999	Priority date *(day/month/year)* 05/09/1998
International Patent Classification (IPC) or national classification and IPC G01G9/00		
Applicant COLE, Michael		

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1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 10 sheets, including this cover sheet.

   ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of 4 sheets.

3. This report contains indications relating to the following items:

   I ☒ Basis of the report
   II ☐ Priority
   III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   IV ☒ Lack of unity of invention
   V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations suporting such statement
   VI ☐ Certain documents cited
   VII ☒ Certain defects in the international application
   VIII ☒ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
25/02/2000	05.12.2000
Name and mailing address of the international preliminary examining authority: European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Grand, J-Y Telephone No. +49 89 2399 2472

I. Basis of the report

1. This report has been drawn on the basis of *(substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments (Rules 70.16 and 70.17).)*:

Description, pages:

1-12	as originally filed			

Claims, No.:

1-22	as received on	06/11/2000	with letter of	03/11/2000

Drawings, sheets:

1/4-4/4	as originally filed			

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

   These elements were available or furnished to this Authority in the following language: , which is:

   - ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
   - ☐ the language of publication of the international application (under Rule 48.3(b)).
   - ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

   - ☐ contained in the international application in written form.
   - ☐ filed together with the international application in computer readable form.
   - ☐ furnished subsequently to this Authority in written form.
   - ☐ furnished subsequently to this Authority in computer readable form.
   - ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
   - ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

   - ☐ the description, pages:
   - ☐ the claims, Nos.:

☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

   (Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

IV. Lack of unity of invention

1. In response to the invitation to restrict or pay additional fees the applicant has:

   ☐ restricted the claims.

   ☐ paid additional fees.

   ☐ paid additional fees under protest.

   ☐ neither restricted nor paid additional fees.

2. ☒ This Authority found that the requirement of unity of invention is not complied and chose, according to Rule 68.1, not to invite the applicant to restrict or pay additional fees.

3. This Authority considers that the requirement of unity of invention in accordance with Rules 13.1, 13.2 and 13.3 is

   ☐ complied with.

   ☒ not complied with for the following reasons:
   see separate sheet

4. Consequently, the following parts of the international application were the subject of international preliminary examination in establishing this report:

   ☐ all parts.

   ☒ the parts relating to claims Nos. 1-22.

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-22
	No:	Claims	
Inventive step (IS)	Yes:	Claims	
	No:	Claims	1-22

Industrial applicability (IA)	Yes:	Claims	1-22
	No:	Claims	

2. Citations and explanations
   see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:
see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:
see separate sheet

R Item IV

Lack of unity of invention (Rule 13(1) PCT).

i) The common concept linking together the independent **claims 1, 11, 19 and 21** concerns the evaporation of liquid to be obtained by rotating in a low pressure chamber.

ii) However, this common concept is not novel, see the document **D1** (= DE-A-19 518 540 (see the abstract)).

Hence, the examining authority considers that the following two distinct and separate groups of claimed inventions are not linked as to form a single general inventive concept:

1- A first invention as claimed in **claims 1, 11 and 21** comprising means for controlling weight during evaporation of sample in centrifugal evaporators.

This feature is provided in order to enable the weight of a sample in a centrifugal evaporator to be continuously and accurately measured during operation.

2- A second invention as claimed in **claim 19** comprising for reducing imbalancing in a centrifuge.

This feature is provided in order to enable the operation of a centrifugal evaporator to continue despite a considerable imbalance of forces.

Thus, the application does not meet the requirements of unity because the two above inventions share neither the same nor any corresponding special technical features forming a contribution over the prior art within the meaning of Rule 30 EPC. Moreover, the above problems and solutions are independent of each other, i.e. they do not form a linked series of problems and solutions. Consequently, **claims 1, 11, 19 and 21** do not satisfy the requirements of unity of invention, Rule 13(1) PCT.

Re Item V

Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement.

Reference is made to the following documents:

D1 = DE-A-19 518 540

D2 = EP-A-0 128 590

D3 = DE-A-4 211 760

D4 = US-A-3 692 236

a) Inv ntion 1

i) Lack of inventive step

The present application does not meet the requirements of Article 33(1) PCT because the subject-matter of the following claims does not involve an inventive step in the sense of Article 33(3) PCT.

Independent method claim 1

i) The closest prior art is given by the arrangement of **D1** disclosing
- a method of evaporating a liquid sample contained in a sample holder which is mounted within a chamber and rotated by a rotor therein during the evaporation so that centrifugal force is exerted on the contents of the sample holder during the process whilst a pressure below atmospheric is maintained in the chamber in manner known per se, so as to leave as a residue any solid material dissolved or otherwise mixed in the liquid forming the sample (see the abstract).

Thus, all the features of the preamble of **claim 1** are known from **D1**.
Furthermore, the step of heating the sample during evaporation in the chamber to increase the rate of evaporation is also known from **D1** (see the abstract and col. 6, l. 8-21).

ii) The independent **claim 1** is distinguished therefrom in the steps of
- mounting a transducer to monitor the force acting on the sample holder relative to the rotor when rotating at a given speed; and
- obtaining a force signal therefrom, supplying the force signal to a computer means, programming the computer means to compute a value equivalent to the centrifugal force exerted on the sample holder due to rotation of the rotor at the given speed, further programming the computer means to compute a weight value from the force signal using the computed centrifugal force, and further programming the computer means to generate a control signal for controlling the evaporation process in dependence on the computed weight value and controlling the supply of heat to the sample in dependence on the computed weight value.

The transducer together with the computer means are used to measure and process the radial force generated by the centrifugal force of the sample and calculate the mass from it.

However, the use of such a transducer together with processing means is known from **D2** (see "load cell (818)" in fig. 21 and p. 18, l. 12-22), wherein the radial displacement force apparatus permits to determine the weight of the sample in the sample holder, as

providing the same advantages as in the present application. It would therefore be obvious to the skilled person to consider the possibility of using a transducer in the arrangement of **D1** in order to obtain a desired read-out of the weight of the sample in the sample holder as the liquid is being evaporated, thus arriving at an apparatus according to **claim 1**.

Furthermore, the use of processing means for controlling the supply of heat to the sample in dependence on the computed weight value is an obvious possibility suggested by the document **D1** (see col. 7, l. 3-18), wherein the processing means (see "Steuereinrichtung (63)") are programmed to control the supply of heat to the sample (see "einstellung der Leistung des Mikrowellengenerators") in dependence on experimental parameters, which parameters could be e.g. the result of a calculation and particularly a computed weight value, in order to enable the weight of a sample in a centrifugal evaporator to be continuously and accurately measured during evaporation. Thus the subject-matter of **claim 1** does not involve an inventive step and does not satisfy the criterion set forth in Article 33(3) PCT.

Independent apparatus claim 11

It is noted that the independent apparatus **claim 11** corresponds to the independent method **claim 1** in that for every method step of **claim 1** a corresponding structural feature is defined therein.

Therefore with respect to the documents **D1** and **D2**, according to the argumentation above, also the independent **claim 11** does not involve an inventive step and does not satisfy the criterion set forth in Article 33(3) PCT.

Independent method claim 21

The independent method **claim 21** is distinguished from the independent method **claim 1** in that a stored weight value corresponding to the empty weight of the sample holder is <u>stored</u> in computer means and <u>deducted</u> from the computed weight value.

The stored weight value is used to calibrate the measurement by deducting the weight value of the empty sample holder to the computed weight value.

However, in view of paragraph above concerning **claim 1** and general common knowledge, the skilled person would regard it a normal design procedure to calibrate the apparatus in order to obtain a desired read-out of the weight of the sample in the sample holder. Thus, the subject-matter of **claim 21** does not involve an inventive step and does not satisfy the criterion set forth in Article 33(3) PCT.

Depend nt claims 2-10, 12, 14-18 and 22

These claims do not appear to contain any additional features which, in combination with the features of any claim to which they refer, meet the requirements of the PCT with respect to inventive step since they do not contain any additional features which go beyond normal design procedures.

Dependent claim 13

The feature "the first mentioned transducer is a load cell" is known from the document **D2** (see "load cell (818)").

b) Invention 2

i) Lack of inventive step

The present application does not meet the requirements of Article 33(1) PCT because the subject-matter of the following claims does not involve an inventive step in the sense of Article 33(3) PCT.

Independent apparatus claim 19

The closest prior art is given by the arrangement disclosed in the document **D4**, wherein an apparatus comprising
- a rotor (see "shaft (18)") mounted therein for rotation in use about a generally vertical axis;
- a drive means (see "drive motor (20)") for rotating the rotor;
- at least two sample holders (see "holders (34, 34(A))") mounted on the rotor;
- each sample holder being pivotal in use about a generally horizontal axis in a radial manner relative to the axis of rotation (see col. 2, l. 66 - col. 3, l. 4);
- a bearing raceway (see "cavity, annular chambers and solid bodies" in fig. 2) incorporating;
- a plurality of ball bearings (see col. 4, l. 13-14, col. 5, l. 5-13 and fig. 2) which do not fully occupy the circumferential extent of the raceway and which in rotation are automatically distributed around the raceway to counteract any imbalancing forces;
- the raceway being mounted to the rotor or a spindle driving the rotor, thereby to reduce any imbalance caused during the spinning of the rotor as result of differential evaporation of liquids from the sample holder (see fig. 2).

The independent apparatus **claim 19** is distinguished therefrom in that the apparatus comprises a vacuum chamber.

The idea of using a vacuum chamber in centrifugal evaporators is generally well-known in the art and particularly from the document **D1** (see abstract and figures) and from the background of the invention acknowledged by the applicants on page 1 of the present application wherein centrifugal evaporators comprising vacuum-tight chamber are well-known in the art.
The vacuum chamber of **D1** is employed to evacuate evaporated solvent from the centrifugal evaporator. However, the skilled person would realise that a further advantage of a vacuum chamber is that any environmental effect on the residue after evaporation of the solvent is prevented. The enclosure of a sample in a vacuum chamber ensures that no medium is present in the immediate vicinity of the sample which could react with the residue and cause the decomposition thereof.
It would therefore be obvious to the skilled person to consider the possibility of using a vacuum chamber in the arrangement given by the document **D4** in order to avoid environmental effects on the residue after evaporation of the solvent thus arriving at an apparatus according to **claim 19.**
Thus the subject-matter of the independent apparatus **claim 19** does not involve an inventive step and does not satisfy the criterion set forth in Article 33(3) PCT.

Dependent claim 20
In this claim, a slight constructional change in the device of **claim 19** is defined which comes within the scope of the customary practice followed by persons skilled in the art, especially as the advantages thus achieved can readily be foreseen. Consequently, the subject-matter of this claim also lacks an inventive step.

Re Item VIII
Certain observations on the international application.
a) Invention 1
i) The 2 definitions of the invention given in the independent method **claims 1 and 19** are such that the claims as a whole are not concise, contrary to article 6 PCT.

ii) The description has not been brought into conformity with the remaining independent claims (cf. article 6 PCT).

Re Item VII

Certain defects in the international application.

a) Invention 1

i) Not all the independent claims have been cast in two-part form (Rule 6.3(b) PCT).

ii) The documents **D1, D2, D3 and D4** have not been identified in the description (Rule 5.1 (a)(ii) PCT.

iii) Reference signs in parentheses have not been inserted in all the claims (Rule 6.2(b) PCT).

CLAIMS

1. A method of evaporating a liquid sample contained in a sample holder which is mounted within a chamber (14) and rotated by a rotor (5A, 5B) therein during the evaporation so that centrifugal force is exerted on the contents of the sample holder during the process whilst a pressure below atmospheric is maintained in the chamber in manner known per se, so as to leave as a residue any solid material dissolved or otherwise mixed in the liquid forming the sample, characterised by the steps of:

mounting a transducer (19) to monitor the force acting on the sample holder relative to the rotor (5A, 5B) when rotating at a given speed and obtaining a force signal therefrom, supplying the force signal to a computer means (54), programming the computer means to compute a value equivalent to the centrifugal force exerted on the sample holder due to rotation of the rotor at said given speed, further programming the computer means to compute a weight value from the force signal using the computed centrifugal force, and further programming the computer means to generate a control signal for controlling the evaporation process in dependence on the computed weight value; and further comprising the steps of heating the sample during rotation in the chamber to increase the rate of evaporation, and controlling the supply of heat to the sample in dependence on the computed weight value.

2. A method as claimed in claim 1, further comprising the steps of mounting a second transducer to monitor the speed of rotation of the rotor, obtaining a speed signal therefrom, and supplying the speed signal to the computing means for computing said weight value.

3. A method as claimed in claim 1 or claim 2, wherein the computing means is adapted to rotate with the rotor.

4. A method as claimed in any one preceding claim, wherein the computing means is

programmed to convert the transducer signals into a form suitable for transmission to an external receiver.

5. A method as claimed in claim 4, wherein the computing means converts the transducer signals into digital signals by which a carrier signal is modulated to effect the said transmission.

6. A method as claimed in any one of claims 1 to 5, wherein the force and speed signals are produced continuously and the weight and centrifugal force values are continuously computed therefrom.

7. A method as claimed in claim 6, wherein the computing means has stored therein a value equivalent to the weight of the sample holder, and is further programmed to compute a value equivalent to the weight of the contents of the holder by deducting from the computed weight value a value equivalent to the known weight of the sample holder.

8. A method as claimed in any one preceding claim, wherein the computer means computes the rate of change of the computed weight value.

9. A method as claimed in any one preceding claim, comprising the step of controlling the supply of heat in dependence on the computed rate of change of weight value.

10. A method as claimed in claim 9, wherein the supply of heat is reduced as the rate of change of weight with time starts to decline, and the evaporation process is terminated when the rate of change drops to zero, indicating that the sample is dry.

11. Apparatus for evaporating a sample comprised of solid material dissolved or suspended in a liquid, comprising a vacuum chamber, a rotor therein, drive means for rotating the rotor relative to the chamber, a sample holder for containing the sample connected to the rotor, transducer means associated with the sample holder and the rotor for generating a force signal indicative of the centrifugal force acting on the sample holder as it is rotated at a given speed, and means for transmitting transducer signals to

computing means programmed to convert the signal at any instant to a computed value proportional to weight, the computing means being further programmed to generate a process control signal for controlling the evaporation process in the chamber.

12. Apparatus as claimed in claim 11, further comprising second transducer means associated with the rotor for generating a speed signal corresponding to the speed of rotation of the rotor, the speed signal being transmitted to the computing means for computing a weight value.

13. Apparatus as claimed in claim 11 or claim 12, wherein the first mentioned transducer is a load cell.

14. Apparatus as claimed in claim 11 or claim 12, wherein the first mentioned transducer is a strain gauge.

15. Apparatus as claimed in claim 11 or claim 12, wherein the sample holder is movable relative to the rotor and the force transducer is a position sensor adapted to produce a signal indicating the position of the sample holder relative to the rotor, as determined by the instantaneous centrifugal force acting on the sample holder, causing it to move relative to the rotor.

16. Apparatus as claimed in claim 15 wherein resilient means resists the movement of the sample holder relative to the rotor.

17. Apparatus as claimed in any one of claims 11 to 16, wherein a plurality of sample holders are mounted on the rotor and a force transducer is provided for at least selected ones the holders.

18. Apparatus as claimed in any one of claims 11 to 17, wherein a mechanical device is attached to the rotor or a spindle on which the rotor is carried and by which it is rotated, which automatically adjusts its centre of mass in response to out-of-balance forces acting on the rotor due to differential evaporation of samples.

19. Apparatus comprising a vacuum chamber, a rotor mounted therein for rotation in use about a generally vertical axis, a drive means for rotating the rotor, at least two sample holders mounted on the rotor, each sample holder being pivotal in use about a generally horizontal axis in a radial manner relative to the axis of rotation, a bearing raceway incorporating a plurality of ball bearings which do not fully occupy the circumferential extent of the raceway and which in rotation are automatically distributed around the raceway to counteract any imbalance forces, the raceway being mounted to the rotor or a spindle driving the rotor, thereby to reduce any imbalance caused during the spinning of the rotor as result of differential evaporation of liquids from the sample holder.

20. Apparatus as claimed in claim 19, wherein the ball bearings are formed from a high density material such as Tungsten or depleted Uranium.

21. A method of measuring the weight of a liquid sample in a sample holder attached to a rotor in a vacuum chamber of an evaporating centrifuge, comprising the steps of mounting a transducer to monitor the force acting on the sample holder relative to the rotor during rotation, supplying a force signal to a computing means having stored therein a stored weight value corresponding to the empty weight of the sample holder, the computing means being programmed to convert the force signal to a weight value for a given speed of rotation of the rotor, the computing means being further programmed to deduct from the computed weight value said stored weight value.

22. A method as claimed in claim 21, further comprising the steps of monitoring the speed of rotation of the rotor, and supplying a speed signal to the computing means for computing said weight signal.

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PATENT COOPERATION TREATY

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INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference DB/C1071.01/C	**FOR FURTHER ACTION** see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. PCT/GB 99/01790	International filing date *(day/month/year)* 07/06/1999	(Earliest) Priority Date *(day/month/year)* 05/09/1998
Applicant COLE, Michael		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

[X] It is also accompanied by a copy of each prior art document cited in this report.

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1. **Basis of the report**
   a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.
      [ ] the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).
   b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :
      [ ] contained in the international application in written form.
      [ ] filed together with the international application in computer readable form.
      [ ] furnished subsequently to this Authority in written form.
      [ ] furnished subsequently to this Authority in computer readble form.
      [ ] the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
      [ ] the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

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2. [X] **Certain claims were found unsearchable** (See Box I).
3. [X] **Unity of invention is lacking** (see Box II).

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4. With regard to the **title**,
   [X] the text is approved as submitted by the applicant.
   [ ] the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,
   [X] the text is approved as submitted by the applicant.
   [ ] the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No. 1
   [X] as suggested by the applicant.
   [ ] because the applicant failed to suggest a figure.
   [ ] because this figure better characterizes the invention.
   [ ] None of the figures.